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MICROSTRUCTURE AND MECHANICAL PROPERTIES OF PRODUCT FROM EXTRUSION CONSOLIDATED 7075 Al PREALLOYED POWDER

WESTINGHOUSE ELECTRIC CORPORATION
ADVANCED ENERGY SYSTEMS DIVISION
BEULAH ROAD
PITTSBURGH, PENNSYLVANIA 15235

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AIR FORCE MATERIALS LABORATORY
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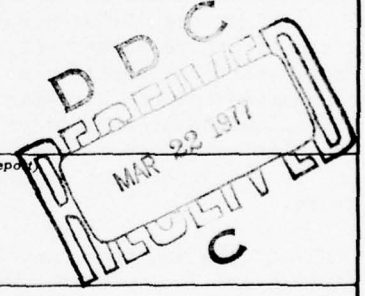

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Microstructural changes occurring during the extrusion-consolidation of prealloyed 7075 stock were investigated and compared with those occurring in the wrought stock (from which the powder was produced) processed in the same manner as the powder. Emphasis is placed on two areas:

1. The effects of processing variables (starting material condition, processing temperature and amount of deformation) on the microstructure of the processed material.

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2. Correlation of the product microstructures with product room tensile properties after a final standard T6 heat treatment.

The microstructure of the extrusion-consolidation powder product differed in several respects from that of the wrought stock product extruded by the same processing conditions. The latter had a uniform microstructure while the former contained a marked inhomogeneity of both the distribution of the precipitate and the size of the substructure or grain structure (from one powder particle to another) and the presence of oxide boundaries on deformed powder particles. The differences are attributed to the following characteristics of the starting materials:

1. The powder consisted of chilled cast particles coated with oxide films and containing elements (such as chromium) in various degrees of super-saturated solution in different particles.

2. The wrought material was homogeneous without any apparent oxide films.

The microstructure within the powder particles of the extruded powder product approached that of the wrought material as the processing variables converted the particle cast structure to the same original condition as the wrought material. However, none of the processing variables were beneficial in the removal of the oxide film around the powder particles.

Extrusion-consolidated 7075 powder which had not been given a long time high temperature anneal yields lower tensile strengths than the extruded ingot product. This difference is the result of the occurrence of an equiaxed recrystallized grain structure rather than a hot work substructure. The powder product also shows inferior ductility as a result both of the oxide film at the prior particle boundaries and of inter-particle voids in the product which arise from incomplete consolidation at the lowest extrusion ratios and from excessive deformation at the high extrusion ratios. In both cases, these voids were more numerous after the T6 heat treatment implying limited bonding at prior particle interface surfaces.

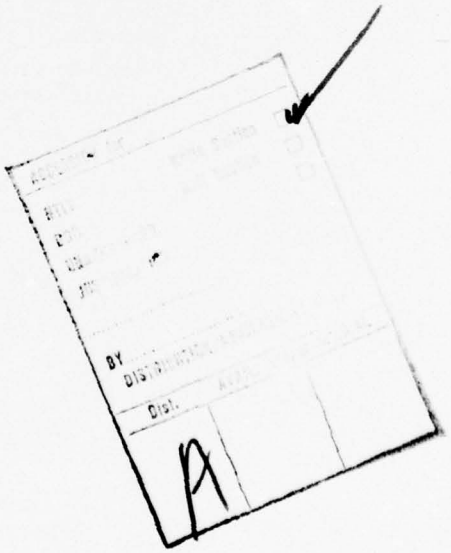
Use of the separate consolidation and extrusion operations at extrusion ratios of 10:1 and 20:1 yields powder product with a fine subgrain and recrystallized grain size. The chromium containing these two-step processed material similar to that in the ingot product. Comparison of the powder product from the two-step process with that from the one-step process suggests that the formation of this precipitate has a significant effect on limiting the grain size resulting from primary recrystallization. The tensile strength of the two-step processed powder material is comparable to that of the ingot product but the ductility is significantly lower.

FOREWORD

This report was prepared by the Westinghouse Electric Corporation, Astronuclear Laboratory, Advanced Energy Systems Division, Pittsburgh, Pennsylvania 15235, under USAF Contract F33615-74-C-5059. The contract was initiated under Project No. 7351, "Metallic Materials", Task No. 735108, "Processing of Metals", and was administered under the direction of the Metals and Ceramics Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio with Mr. A. M. Adair (AFML/LLM) as Project Engineer.

This report covers work performed from 16 December 1973 to 1 July 1976, by Messrs. D. J. Abson, F. J. Gurney of the Advanced Energy Systems Division, Westinghouse Electric Corporation, and V. DePierre of the Processing and High Temperature Materials Branch, Metals and Ceramics Division (AFML/LLM).

This report was submitted by the authors 15 September 1976.



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SECTION I

INTRODUCTION

United States Air Force interest in powder metallurgy stems largely from the potential benefits obtainable by the use of metal powder, instead of ingot material, for the production of high strength metal products. Exploitation of these benefits requires an intelligent selection of processing methods and parameters, based on an understanding of both the mechanics and metallurgy of the operation. Specifically, these benefits include more economical and/or better properties of the final product. The better properties are possible because inherently the metal powders have finer grains and less macro-segregation than cast ingot materials. These powder characteristics may provide the following advantages over ingot material for metalworking operations:

1. Greater resistance to fracture during forming.

2. Higher processing temperatures without incipient melting or hot shortness.

3. Lower deformation loads for hot working operations.

4. Less requirement for a homogenization treatment with the possibility of eliminating such a treatment.

Additionally, the use of the powder route may also permit the following results:

1. Products with better properties (such as toughness, crack growth resistance and resistance to stress-corrosion cracking) because of the powder fine grain and homogeneity.

2. Introduction of compositional variables which result in significant variation during the solidification of large ingots but remain in super-saturated solid solution during rapid cooling of the metal powder and enhance the service properties of the product.

3. Synthesis of composite alloys by blending different powders to provide desirable combinations of properties not obtainable in single alloys.

In an earlier publication[1], the mechanics of metal powder processing methods were discussed in detail with 7075 aluminum alloy powder as the model material and extrusion-consolidation as the process. In that work, general variations in the microstructure were also discussed. The primary purpose of this report is to discuss in greater detail the microstructural changes occurring during the extrusion-consolidation of prealloyed 7075 powder and to compare these changes with those occurring in the wrought stock (from which the powder was produced) processed in the same manner as the powder. Emphasis is placed on the following areas:

1. The effects of processing variables (such as starting material condition, processing temperature and amount of deformation) on the microstructure of the processed material.

2. Correlation of the product microstructures with product room temperature tensile properties after a final standard heat treatment.

SECTION II

EXPERIMENTAL PROCEDURES

2.1. Material

The material used in this study was obtained as a single heat lot of wrought 3-inch (0.076m) diameter bars of commerical 7075-T6 aluminum alloy. The chemical analysis of the lot is given in Table I. A portion of the material was machined into extrusion billets 3.00 inches in diameter and 6 inches long (0.076m x 0.15m). The remainder was converted to powder by the rotating electrode process.

2.2. Material Preparation

Prior to shipment, the powder was encapsulated under argon in small metal containers. All further handling of the powder was done under an argon atmosphere. A sieve analysis of the powder is given in Table II. The logarithm of the particle size was nearly normally distributed with a median particle size of 132 x 10^{-6}m, approximately 110 mesh. Chemical composition of the powder is also listed in Table I.

Prior to consolidation, the metal powder was encapsulated in 6061 aluminum alloy containers 3.00 inches in diameter and 6.0 inches long (0.076m x 0.15m) which were evacuated and sealed as described in Appendix I. The pour density of the powder was approximately 60 percent.

2.3. Processing

The machined wrought billets and powder-filled containers were processed in the AFML 700 Ton (6.2 x 10^{4}N) Horizontal Extrusion Press. Details of the extrusion procedure are given in the previous publication[1] the previous processing conditions for all billets are presented in Table III and Table IV. Three different processes were used for the powder containing billets.

Process (A) Consolidation by compaction against "blank" tooling at a pressure of 180 ksi (1.24 x 10^{-9}Nm^{-2}) for 60 seconds after an 800°F (700°K) 2 hour preheat on the powder container.

Process (B) One-step extrusion-consolidation after 2 hour preheat at the desired temperature.

Process (C) Two-step extrusion-consolidation consisting of Process (A) cooling the consolidated billet to room temperature, remachining the billet and then extruding as related in Process (B).

Wrought billets were processed as described in Table III for comparison of extruded wrought products with powder extrusions.

2.4. Tests

The following tests were made on the metal powder and extruded products:

2.4.1 Chemical Analyses - on selected powder extrusions to determine the

effects of processing conditions on chemical composition of the processed material. Results were essentially the same as for the as-received powder as listed in Table I.

2.4.2 Optical Microscopic Examinations - The specimens were electro-polished using a solution consisting of 60 ml. perchloric acid, 350 ml. butyl cellosolve and a potential of 28 volts applied for two periods each of 9 seconds for electro-polishing. The polished specimens were etched in Keller's etch.

Powder material was examined in each stage of the processing operation and compared to wrought product. Powder specimens, after various heat treatments simulating prior-extrusion temperature time conditions, were examined to establish the microstructure present immediately before extrusion and to serve as a basis for determining the effects of the one-step extrusion-consolidation processing conditions on the powder product microstructure. Specimens from the hot consolidated billet (No. 4911, Table III) were examined to establish the prior extrusion microstructural conditions of material processed by the two-step (Process C). Longitudinal sections of the extruded products in the as-extruded and in the heat-treated conditions were examined.

2.4.3 Electron Metallography - The thin film electron microscopy examinations for the study were limited to material extruded at 800°F (700°K). Longitudinal slices for electron metallography were ground from 0.04 inches (0.001m) to 0.02 inches (0.0005m) thick from specimens taken from the central axis of the extrusion. Discs of 0.13 inches (0.003m) in diameter were punched from the slices and were jet-machined to performation in a solution consisting of 2 ml hydrochloric acid, 50 ml nitric acid and 50 ml methanol. The solution was used at room temperature with a potential difference of approximately 20 volts. The foils were examined in a JEM 200A electron microscope operating at 200 KV.

2.4.4 X-Ray and Electron Microprobe Analyses - As-received powder and longitudinal sections of extruded bars were subjected to x-ray and electron microprobe analyses x-ray images gave information on principal constituents of second-pahse particles. Supplemental information was obtained both by line scans across powder particle cell walls or across selected extruded powder particles, and also by spectral scans at specific locations.

2.4.5 Room Temperature Tensile Tests - A piece of extruded rod of length sufficient for two tensile test bars was cut from the mid-length portion of each extrusion. The specimens were rough-machined from the central portion of each piece to 0.50 inches (0.013m) diameter or where possible to 1.0 inches (0.025m) diameter so that quench rate effects could be controlled during later processing. A standard T6 heat treatment was given to all the rough-machined rods. This treatment consisted of 1 hour at 870°F (749°K), a quench into warm water at 175°F (349°K) followed by a 24 hour hold at room temperature and aging at 250°F (394°K) for 24 hours. Standard type R-3 tensile test specimens of 0.252 inches (.0064m) gage diameter were machined from the heat treated rods and tested at room temperature in a 10,000 lb. (4.45×10^4N) Instron testing machine at a cross-head speed of 0.05 ipm (2.1×10^{-5}ms^{-1}).

SECTION III

RESULTS

3.1. Chemical Compositions

As noted in Table I, the only major differences in composition of the wrought bar and the as-received ans as-processed metal powder used in this investigation are in the oxygen content. The metal powder as-received has a higher oxygen content than the wrought bar. No increase in the oxygen content was apparent in the metal powder after processing operations performed in this investigation.

3.2. Microstructure of Powder Before Extrusion

Optical microscopic examination of the as-received powder revealed a fine ($\simeq 5 \times 10^{-6}$m) cellular or dendritic cast structure (Figure 1a). Little change occurred in the structure after heating the powder for two hours at temperatures up to and including 700°F (644°K). The cell structure had almost disappeared in the powder after treatment at 800°F (700°K) for two hours. The boundaries of several grains were visible in each powder particle, Figure 1b, after heating at 900°F (756°K) for one hour. These tests established the microstructural condition of the metal powder before the one-step extrusion-consolidation processing, Process (B).

The optical microstructure, Figure 2, of hot-consolidated powder, Process (A), showed the powder particles have an equiaxed shape with clearly discernable boundaries and complete elimination of voids between the particles. The cast microstructure, Figure 1a, of the powder has disappeared almost completely as a result of the hot consolidation treatment. This condition represents the microstructure of metal powder before the two-step extrusion-consolidation processing, Process (C).

3.3. Microstructure of the Extruded Powder Products

3.3.1 Optical Microstructure of Air-Cooled One-Step Extrusion-Consolidated Products - The as-cast structure was not completely eliminated from the powder billets processed at temperatures up to 700°F (644°K) but had almost disappeared after heating to 800°F (700°K) and was not detectable in the microstructure of the extruded products at this and higher temperatures.

All metal powders extruded at 3:1 reduction ratio were visually sound but optical microscopic examination at a magnification of 250X showed voids and poor interparticle bonding. The 6:1, 10:1 and 20:1 products in the as-extruded condition were sound both visually and under microscopic examination. However, after the T6 heat treatment, these products contained small voids along the powder particle boundaries and at interparticle triple points. At an extrusion ratio of 40:1, the as-extruded product had a large number of cracks along interparticle boundaries.

Except for the appearance of small voids after the T6 heat treatment, the optical microstructures of the powder products air-cooled after extrusion were almost identical in the as-extruded and in the as-extruded plus T6 heat treatment

condition when viewed at 250X. However, the grain boundaries could be seen most clearly on the heat-treated product. The observed microstructures are summarized later. After extrusion at 800°F (700°K), the product at 3:1 ratio was not recrystallized. However, a substantial portion of the microstructure of the 10:1 extrusion consisted of equiaxed recrystallized grains, 30 x 10^{-6}m in diameter, within each elongated powder particle, Figure 3. Some of these grains occupied the whole width of a powder particle. The proportion of recrystallized grains increased to almost 100 percent as the extrusion reduction was increased through 20:1 and 40:1. The 20:1 - 800°F (700°K) and 10:1 - 900°F (756°K) extrusions had almost identical microstructures both containing grains which had grown across particle powder boundaries, Figure 4.

3.3.2 Optical Microstructure of Air-Cooled Extrusions from Two-Step Process C - In the 10:1 - 800°F (700°K) product from pre-consolidated billet, Figure 6, approximately 30 percent of the microstructure consisted of very fine ($\simeq$ 5 x 10^{-6}mm) recrystallized grains and the remainder contained wrought grains. In the 20:1 - 800°F (700°K) extrusion approximately 30 percent of the microstructure consisted of a wrought structure and 35 percent of large grains whose boundaries crossed powder particle boundaries. The microstructure of the recrystallized portion of this extrusion was similar to that shown in Figure 5 for the 40:1 - 800°F (700°K) extrusion made from the preheat treated (900°F - 6 hour plus air cooling) billet.

3.4. Optical Microstructure from Extruded Wrought Billets

The principle change in the optical microstructure during extrusion of the wrought ingot product was the further elongation of the grains. A few very small ($\simeq$ 5 x 10^{-6}m) recrystallized grains were observed in the 6:1 - 800°F (700°K) product and at higher extrusion ratios. Only at an extrusion ratio of 40:1 at 800°F (700°K) was extensive recrystallization observed in the as-extruded product where elongated recrystallized grains ($\simeq$ 1 x 10^{-3}m long and $\simeq$ 1 x 10^{-4}m wide) were surrounded by a small number of finer ($\simeq$ 3 x 10^{-6}m) equiaxed recrystallized grains; no noticeable change occurred in this microstructure as a result of a T6 heat treatment. The large recrystallized grains occurred principally towards the edges of the extruded bar, Figure 7.

3.5. Thin Foil Microscopy of the Product from the 800°F Extrusions

3.5.1 Ingot Product - Transmission electron microscopy of the ingot product in both the as-extruded and T6 condition revealed a uniform subgrain structure with a subgrain size of $\simeq$ 2 x 10^{-6}m. In both conditions, dislocations were resolvable in some of the boundaries and dislocation tangles were visible within the subgrains. Two types of precipitates are present in the T6 condition: 1) the hardening phase in the form of spherical G. P. zones of η platelets[2] of $\simeq$ 100 x 10^{-10}m diameter and 2) a larger spherical precipitate of 3 x 10^{-8} to 5 x 10^{-6}m diameter which is presumed to be the E-Phase[2,3,4] $Al_{18}Cr_2Mg_3$.

The microstructure from the 10:1, 20:1 and 40:1 ratio extrusions appeared very similar to that shown in Figure 8. The product from the 40:1 ratio extrusion, however, had a few very large elongated substructure-free regions. These regions corresponded to the long recrystallized grains seen under the optical microscope. With the exception of this extrusion, grain boundaries were not conspicuous at any extrusion ratio.

In the as-extruded condition, sub-boundaries are less readily discernable. Profuse amounts of η ($MgZn_2$) with largest dimension of 0.5×10^{-6}, Figure 9, were observed. Observation of the E-Phase in the as-extruded condition is difficult because of the masking effect of the η phase.

3.5.2 Powder Product - Transmission electron microscopy of the consolidated powder product revealed several differences from that of the ingot product. The most noticeable of these were the inhomogeneity of both the distribution of the precipitates and the size of the substructure or grain structure. Observation of particle boundaries showed them to be usually straight with a high density of precipitates in the size range 0.02×10^{-6}m, Figure 10, at the particle boundaries. These boundaries were usually straight and usually marked a discontinuity of microstructure from particle to particle. Since these smaller precipitates were seen only at the particle boundaries and since they were almost transparent to the electron beam, it seems likely that they are fragmented oxide particles.

The remaining precipitates were essentially the same as those observed in the ingot product. The E-Phase precipitates in the powder product were less profuse and smaller ($\simeq 0.01 \times 10^{-6}$m to 0.03×10^{-6}m) than those in the ingot product.

Periodic variation in the density of distribution of $MgZn_2$ precipitates occurred. At certain orientation, these precipitates were seen to occur as distinct rows where a high density of these precipitates occurred, next to regions of almost precipitate-free matrix. The alignment of these rows was parallel to the extrusion direction and the spacing was approximately 10^{-6}m.

3.6. Microstructural Anomaly

In all the extrusion-consolidated products, a few powder particles (less than one percent) behaved differently from the others under processing conditions. These particles were generally fully recrystallized, even in the as-extruded condition; the only exception was in the billet which was simply consolidated where, in the as-consolidated condition, they had not recrystallized. The anamolous particles were distinguished by their lighter color during microscopic examination and, in the low ratio extrusions, by their convex boundaries which indicated that they were softer, at the processing temperature than the surrounding particles.

In the consolidated billet after T6 heat treatment, the grain sizes with these anomalous particles were typically ASTM No. 10 and 11, considerably finer than the grain size of the other powder particles. In the extrusion-consolidated product, grain sizes were usually ASTM No. 5 at the lower extrusion ratios, Nos. 5 and 6 in the 20:1 - 800°F (700°K) product and Nos. 6 and 7 in the 40:1 - 800°F (700°K) product. In the two-step extrusion-consolidated product, these anomalous grains were more difficult to discern in the as-extruded condition.

Electron probe micro-analysis of the anomalous particles revealed that they were depleted slightly in copper, magnesium and zinc. The exact origin of these particles is not definite, but they may be attributable to segregation in the wrought stock from which the powders were produced.

3.7. Room Temperature Tensile Test Results

The variation of strength and ductility with processing temperature at a constant extrusion ratio of 10:1 are shown in Figure 13. Both extrusion compacted powder and extruded ingot stock are examined. The highest strength in the extrusion-compacted powder after T6 heat treatment, resulted from processing in the preheat range of 700°F (644°K) to 800°F (700°K) at preheat temperature of 800°F (700°K) and above, the dendritic structure of the powder particles, Figure 1a, was almost completely absent. This temperature was utilized for processing at a range of extrusion ratios. The reduction in the ductility at 800F (700°K) is associated with the initial disappearance of the cast structure; the increased ductility in the powder product after 900°F (753°K) extrusion is associated with a recrystallized microstructure from this product.

The mechanical property data after extrusion at 800°F (700°K) and T6 heat treatment are shown in Figures 14a and 14b. For the ingot product, both strength and ductility increased slightly with increasing deformation in the range between 3:1 and 20:1. At deformation ratios for the ingot product of 40:1 and higher, the large recrystallized grains develop and a resultant loss of strength and increase in ductility develop. At deformation ratios less than 3:1 for the ingot product, another region of decreased strength and increased ductility is expierenced; the reason for this effect at the low ratios is not available.

The maximum strength conditions for the powder billets consolidated by Process (B) resulted from processing at the 6:1 and 10:1 deformation ratios. At higher ratios, large recrystallized grains occurred and a subsequent reduction in strength and increase in ductility resulted. At still higher ratios, porosity developed in the vicinity of inclusions and second phase particles; this porosity causes a continued decrease in strength and a reversal in the ductility trend thereby resulting in a rather severe reduction in ductility. At the 3:1 deformation ratio, the processing technique resulted in tensile stresses along the axis of the compacted bar and separation at the oxide contaminated particle interfaces.

SECTION IV

DISCUSSION

4.1. The Influence of the Powder Production Technique on the Microstructure

The nature of powder production by the rotating electrode process can influence the chemical homogeneity and size distribution of the powder product. The basic nature of this powder production process is that an arc is established between a cathodic tungsten electrode and one end of a consumable ingot rod of the alloy composition desired for the powder which is the anodic electrode. The anodic ingot electrode is held in a chuck and rotated at a selected number of revolutions per minute. The cathodic tungsten electrode is non-rotating, but is made to move axially to maintain a fixed arc length between the two electrodes. The established arc then causes melting to occur and powder is produced when the surface tension forces between the molten alloy and the solid ingot are overcome by the centrifugal forces resulting from the rotating electrode.

Two important consequences of this type of powder production process should be given attention. The first of these is that different centrifugal forces are experienced over the face of the rotating electrode. A molten region near the outside diameter of the bar face is thrown off more quickly than it would be for a molten region near the axis of the bar.

The second factor is related to the first in that the amount of super-heat that a particular region experiences is dependent upon its time of exposure to the arc. Since the region near the electrode axis is subjected to lower centrifugal forces than regions near the outside diameter, it would be expected that a somewhat longer heating time and thus a higher degree of super-heat would be experienced by material near the center of the electrode face than would be experienced by material from near the outside diameter of the electrode face.

The above discussions are more applicable if the face of the consumable electrode is convex. If the face is concave then the argument must be modified to account for material from the center of the electrode sliding over and mixing with the material from the outer edges of the bar as a globule of molten material is in the process of being thrown from the electrode face.

The extent and variation in the amount of super-heat can be of significance depending on the alloy being melted. In the case of the type 7075 aluminum, the E-Phase, $Al_{18}Cr_2Mg_3$, has a higher melting temperature than the parent material.* However, this fact does not preclude the possibility of this phase being dissolved, either completely or in part, by the matrix material during the time that the matrix becomes molten and before it resolidifies as a powder particle after being thrown from the rotating electrode. This dissolving process could occur at molten matrix temperatures considerably lower than the E-Phase melting temperature, especially since the precipitate size of the E-Phase is very small.

*An attempt in our laboratory to determine the melting temperature of this phase was made on a homogenized cast "button" ingot of slightly off stoichiometric composition. The melting point was not defined exactly, but was determined to be in excess of 1600°F (1140°K), indicating that the E-Phase has high thermal stability in the 7075 matrix.

4.2. Chemical Composition

Since the powder material used in this investigation was produced from the same heat as the ingot stock used for comparison of properties, the only significant difference in chemical composition expected would be in absorbed gases with little change in any of the principle alloying constituents. Data in Table I reveal that this is indeed the case. The increased oxygen pick-up occurs during the production of the powder from the ingot bar by the rotating electrode process and during subsequent handling of the powder prior to consolidation (an inert atmosphere of argon was used during all subsequent handling processes).

In the microstructure examinations, it was revealed that the increased oxygen in the metal powder is concentrated as a film on the powder particles. The film nature of the oxide exerts a profound influence on the processing properties, microstructural characteristics, and product tensile properties of the powder material.

Although the overall compositions of the major alloying elements are similar in both the ingot and powder product, some inhomogeneity of both the distribution of the precipitates and size of both the substructure and grain structure occurred in the powder material. However, electron microprobe analysis did not establish any detectable differences in chemical composition of the particles where these variations occurred and therefore the inhomogeneity in microstructure is apparently the result of the extent to which the alloy constituents are in or out of solution.

4.3. Effect of Processing Variables on Microstructure

The microstructure of the extrusion-consolidated powder product differed in several respects from that of the product extruded from the wrought material by the same processing conditions. The latter had a uniform microstructure while the former contained a marked inhomogeneity of both the distribution of the precipitate and the size of the substructure or grain structure (from one powder particle to another) and the presence of oxide boundaries on deformed powder particles. The differences are attributed to the following characteristics in the starting materials:

(1) The powder consisted of chilled cast particles coated with oxide films and containing elements (such as chromium) in various degrees of super-saturated solution in the aluminum base material in different particles.

(2) The wrought material was homogeneous without any apparent oxide films.

The microstructure within the powder particles of the powder product approached that of the wrought product as the processing variables converted the cast structure of the particle to the same original condition as the wrought material. However, none of the processing variables were beneficial in the removal of the oxide film around the powder particles. In all other respects, the effects of processing variables on the microstructure of the powder product tend to approach the effects on the microstructure of the wrought product.

SECTION V

CONCLUSIONS

The following conclusions are made as the result of the metallurgical and mechanical property evaluation of the consolidated powder and of ingot product of 7075 aluminum.

1. Extrusion of the 7075 Al ingot product at 800°F (700°K) yields a product with a well developed subgrain structure. The cell size is approximately 2×10^{-6}m for deformation ratios of 20:1 and lower; the substructure was found to change only slightly during a T6 heat treatment. Exceptions were noted at higher deformation ratios. Ingot stock extruded at a ratio of 80:1 gives a microstructure of fine equiaxed recrystallized grains which were replaced by very large recrystallized grains during the T6 heat treatment. Extrusion at a 40:1 ratio yielded a duplex as-extruded microstructure. Along the axis of the bar, a zone of approximately one-half the bar diameter was not recrystallized, while the remaining outer portion of the bar yielded a large grained recrystallized product. No significant changes occurred in the microstructure as a result of the T6 heat treatment.

2. Extrusion consolidated 7075 powder product which had not been given a long time high temperature anneal (equivalent to the homogenization treatment of cast product) yields a lower strength than the extruded ingot product. This difference is the result of the occurrence of an equiaxed recrystallized grain structure rather than a hot work substructure. The powder product also shows inferior ductility as a result both of the presence of an oxide film at the prior particle boundaries and of internal voids in the product which arise from incomplete consolidation at the lowest extrusion ratios (3:1) and from excessive deformation at the high extrusion ratios (20:1 and 40:1). These high deformation ratio defects also appear as interparticle defects. In both cases, these defects were more numerous after the T6 heat treatment implying limited bonding at prior particle interface films.

3. Use of the separate consolidation and extrusion operations at extrusion ratios of 10:1 and 20:1 yield product with a finer subgrain and recrystallized grain size. The chromium containing E-Phase precipitate was uniformly distributed in these two-step processed material similar to that from the ingot product. Comparison of the product from the two-step processed material with that from the one-step processed material suggests that the formation of this precipitate has a significant effect on limiting the grain size resulting from primary recrystallization. The tensile strength from the two-step processed material is comparable to that from the ingot product but the ductility is significantly reduced.

TABLE I

Chemical Compositions (Weight Percent-Balance Aluminum)

Identity	Zn	Mg	Cr	Cu	Fe	Si	Ti	O	N
Bar As-Received	5.7	2.3	0.19	1.7	.17	.08	.007	.005	.005
Powder As-Received	5.2	2.4	0.19	1.7	N/E	N/E	N/E	.032	.009

N/E = Not Examined

TABLE II

Sieve Analysis of the Powder

Sieve, U.S. Series	45	50	60	80	120	170	230	325	Pan
Microns	354	297	250	177	125	88	63	44	
% Retained on Screen	.69	2.08	10.17	43.20	32.69	8.19	2.41	.51	.06

TABLE III

Extrusion Parameters[+] for 7075 Powder Billets

A Powder Billets

Temperature	Extrusion Ratio					
°F	3:1	6:1	10:1	20:1	40:1	Blank
500	4629 C-B		4630 C-B			
500	4631 C-6		4632 C-6			
700	4616 C-B		4619 C-6			
700	4618 C-6		4617 C-B			
600			4746 C-6			
750			4909 C-6			
800	4793 C-6		4829*C-6	5059*C-6		
800	4796 C-6	4794 C-6	4678 B-B	4795 C-6	4910 C-6	4911**C-6
800			4679 C-B			
800			4680 C-6			
800			4724(d) C-6			
900	4620 C-B		4621 C-B			
900	4622 C-6		4623 C-6			

+ For key to symbols B-B, C-B and C-6 denoting the type of lubricant, see Table IV

* Preconsolidated, cooled to room temperature, remachined and extruded

** Preconsolidated by compacting the powder billet against a blind die after 2 hour 800°F (700°K) preheat The consolidation was performed by subjecting the billet to a pressure of 180 ksi ($1.24 \times 10^{-9} Nm^{-2}$) for approximately 30 seconds

TABLE IV

Extrusion Parameters for the 7075 Wrought Billets

Temperature	Extrusion Ratio					
°F	3:1	6:1	10:1	20:1	40:1	80:1
800	4993 C-6	4992 C-6	4991 C-6	4990 C-6	4989 C-6	5097 C-6

Notes: (1) All billets were heated for two hours in a furnace preheated to the selected temperature and extruded through 90° conical dies at a ram speed of 0.5 inches per second.

(2) Lubrication conditions are noted as follows:

B-B - No lubrication on billet, container and die.

C-B - Intermediate lubrication Graphite-MoS_2-PbO coating on billet and no lubrication on container and die.

C-6 - Good lubrication, coating on billet and a container and die lubricant consisting of graphite, aluminum and lead-dispersed oil base.

TABLE V

Recrystallization Data for 7075 Powder Billets

Extrusion Temperature °F	Extrusion Ratio				
	3:1	6:1	10:1	20:1	40:1
500°F	Wrought	N/E	Wrought	N/E	N/E
600°F	N/E	N/E	Wrought	N/E	N/E
700°F	Wrought	N/E	Wrought	N/E	N/E
750°F	N/E	N/E	Onset of Recry.	N/E	N/E
800°F	Wrought	20% Recry.	50% Recry.	80% Recry.	95% Recry.
900°F	50% Recry.	N/E	80% Recry.	N/E	N/E

N/E = Not Examined

TABLE VI

Recrystallization Data for 7075 Wrought Billets

Extrusion Temperature °F	Extrusion Ratio 3:1	6:1	10:1	20:1	40:1	80:1
800°F		A few very small ($\simeq 5 \times 10^{-6}$mm Recry. grains)			Extensive Recry. large grains (1×10^{-3}m long and 1×10^{-4}m wide) surrounded by a small number of finer grains ($\simeq 3 \times 10^{-6}$m) at outside edges of extruded bar.	Entirely fine Recry. grains
		T6 Heat Treated			No noticeable change	
						Large grains (3×10^{-3}mm x 5×10^{-4}mm wide)

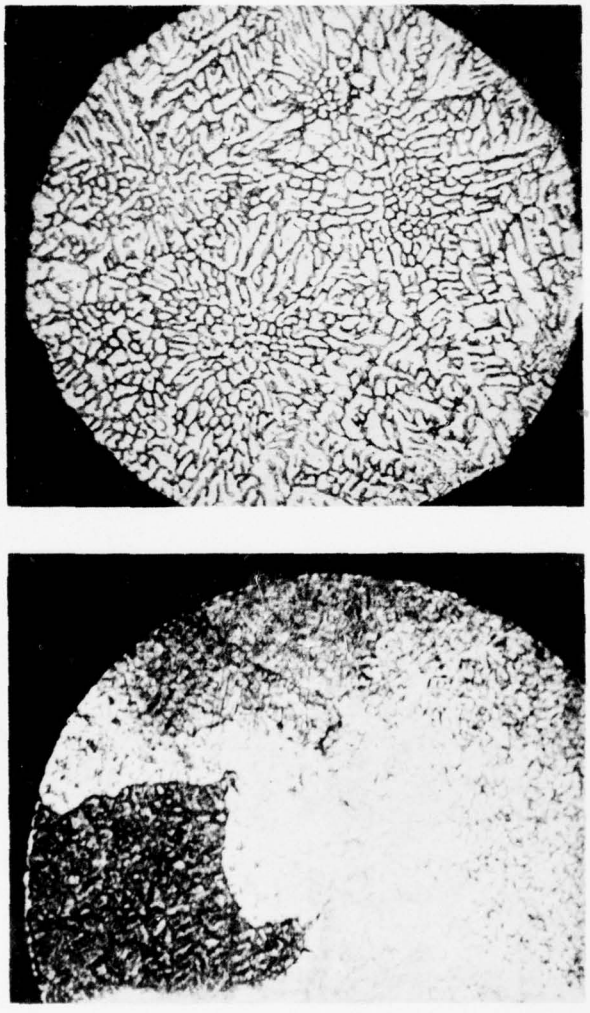

Figure 1. Optical micrographs of powder particles of 7075 alumimum alloy (a) as-received, (b) after a 900°F (756°K) - 1 hour heat treatment.

X600

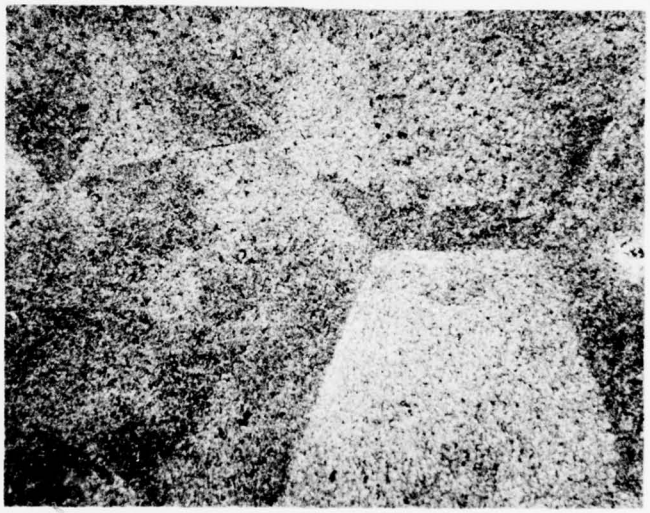

Figure 2. Optical micrograph of the powder consolidated by a pressure of 180 ksi (124 Nm^{-2}) applied for 30 seconds after a 2 hour - 800°F (700°K) preheat. The boundaries are those of the powder particles. Note that the cast structure of Figure 1a has been eliminated but that recrystallization has not occurred. X250

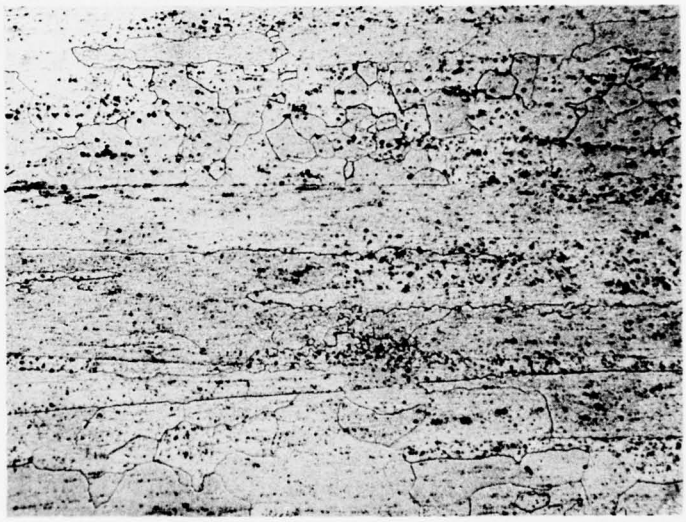

Figure 3. Optical microstructure of extrusion-consolidated powder product (10:1 reduction ratio, 2 hours - 800°F (700°K) extrusion preheat). Note that both recrystallized and non-recrystallized regions are bounded by prior powder particle boundaries. X250

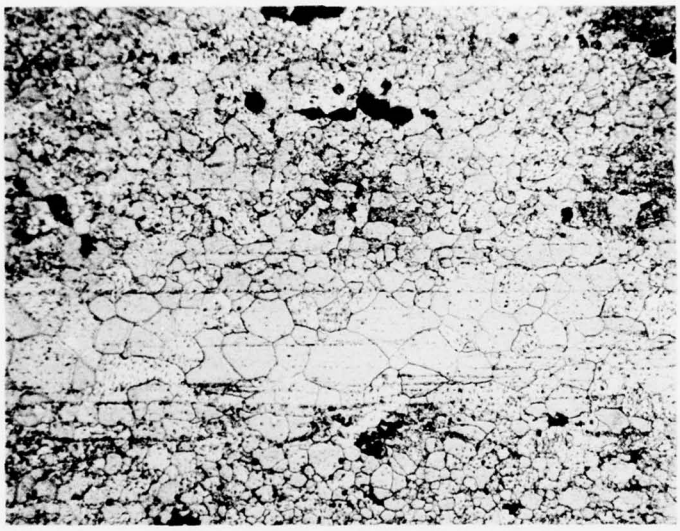

Figure 4. Optical microstructure of extrusion-consolidated powder product (20:1 reduction ratio, 2 hour - 800°F (700°K) extrusion preheat. Note that the recrystallized grains are not confined by the prior powder particle boundaries. X250

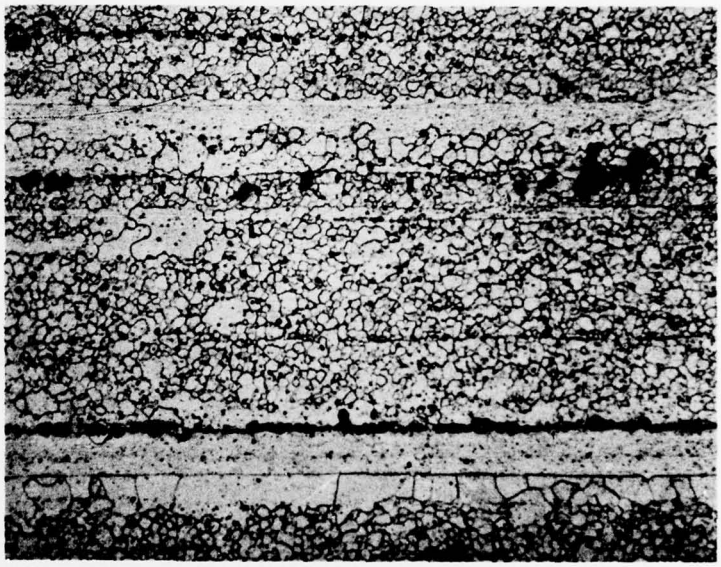

Figure 5. Optical micrograph of extrusion-consolidated powder product which received a 6 hour - 900°F (756°K) heat treatment prior to extrusion at a reduction ratio of 40:1 with a 2 hour - 800°F (700°K) preheat. Note the predominance of the equiaxed (primary) recrystallized grains. X250

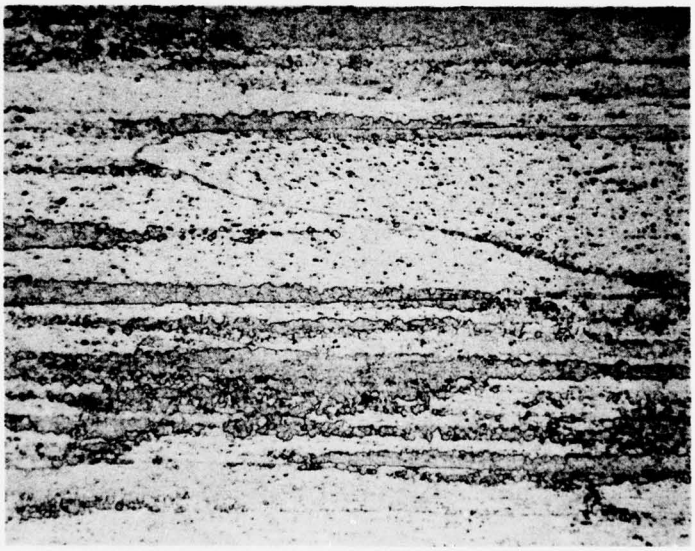

Figure 6. Optical micrograph of powder product extruded at a reduction ratio of 10:1 after prior hot consolidation followed by a 2 hour - 800°F (700°K) preheat and extrusion. X250

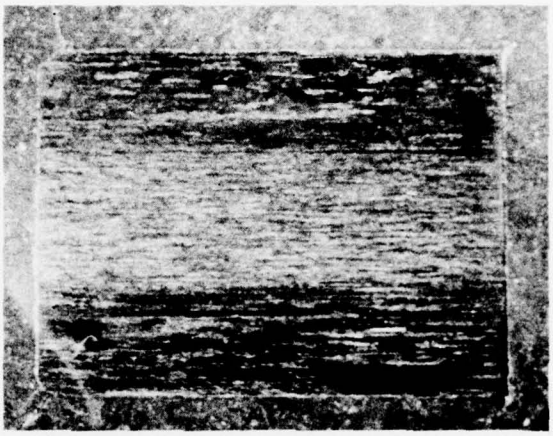

Figure 7. Microstructure of one-half of a bar of ingot stock extruded at 40:1 reduction ratio after a 2 hour - 800°F (700°K) preheat. Note the sharp discontinuity between the wrought material and that which is predominantly recrystallized. X4

Figure 8. Electron micrograph of the ingot product extruded at a 20:1 reduction ratio after a 2 hour - 800°F (700°K) preheat. In the T6 heat condition, showing the presence of a uniform substructure and a uniform distribution of the E-Phase precipitates. X

Figure 9. Electron micrograph of the ingot product extruded at a 20:1 reduction ratio after a 2 hour - 800°F (700°K) preheat. In the as-extruded condition, showing profuse precipitation of laths of η $MgZn_2$. X

Figure 10. Electron micrograph illustrating the discontinuity of microstructure across a prior powder particle boundary in product extrusion-consolidated at a reduction ratio of 10:1 after a 2 hour - 800°F (700°K) preheat. The material is shown in the T6 condition. X9500

Figure 11. Electron micrograph of prior consolidated powder product after extrusion at a reduction ratio of 20:1 followed by a T6 heat treatment. (a) Note the presence of both a uniform distribution of $\simeq 0.05 \times 10^{-6}$m E-Phase precipitates and a powder particle boundary, (b) Recrystallized region containing fine equiaxed grains which are free of substructure, (c) Non-recrystallized region containing a hot work substructure.
X24,000

Figure 12. Electron micrograph of an extrusion-consolidated powder product which had been subjected to a pre-extrusion heat treatment of 6 hour - 800°F and a post-extrusion T6 heat treatment. Note the presence of the fine equiaxed recrystallized grains and the grain-boundary precipitate.
X3300

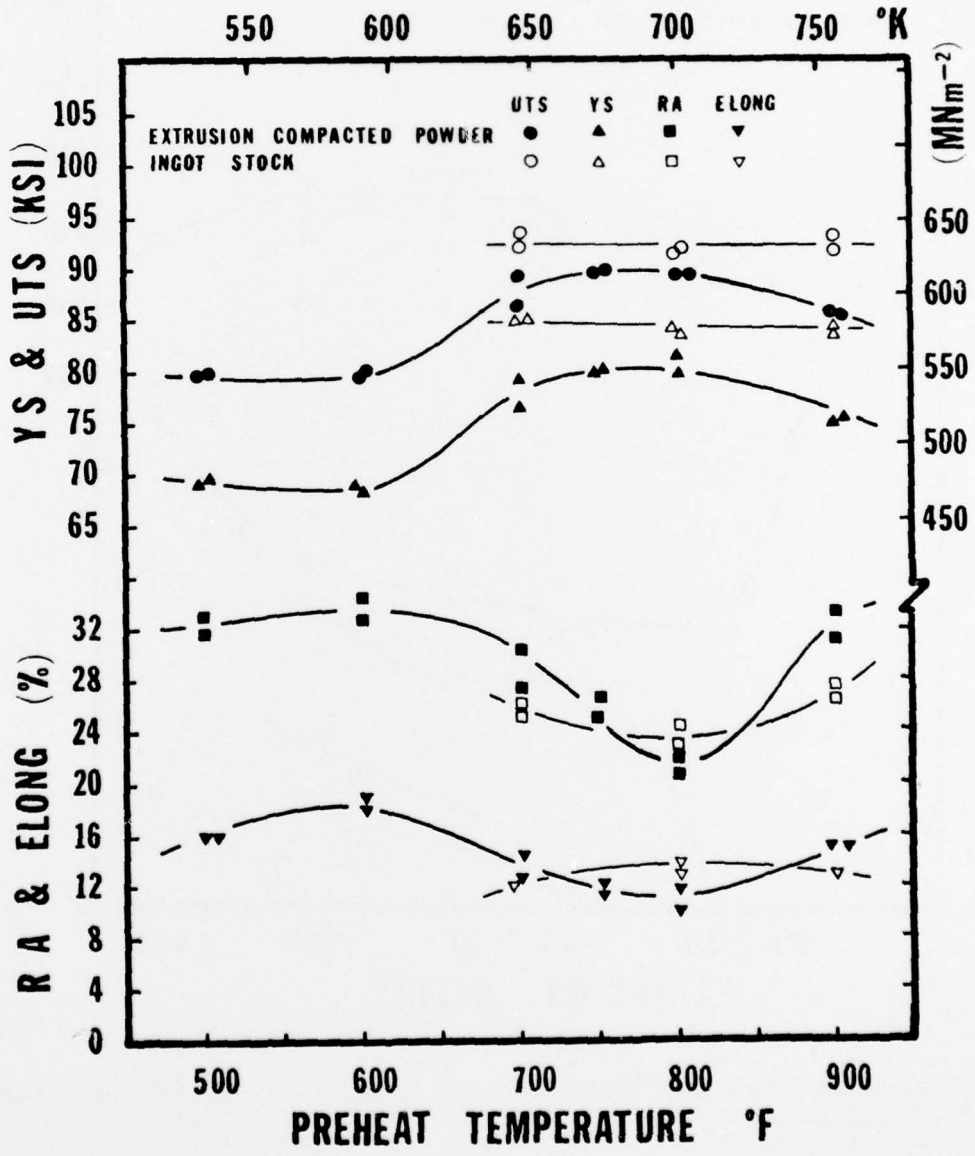

Figure 13. Variation of strength and ductility in both ingot and powder product with extrusion preheat temperature.

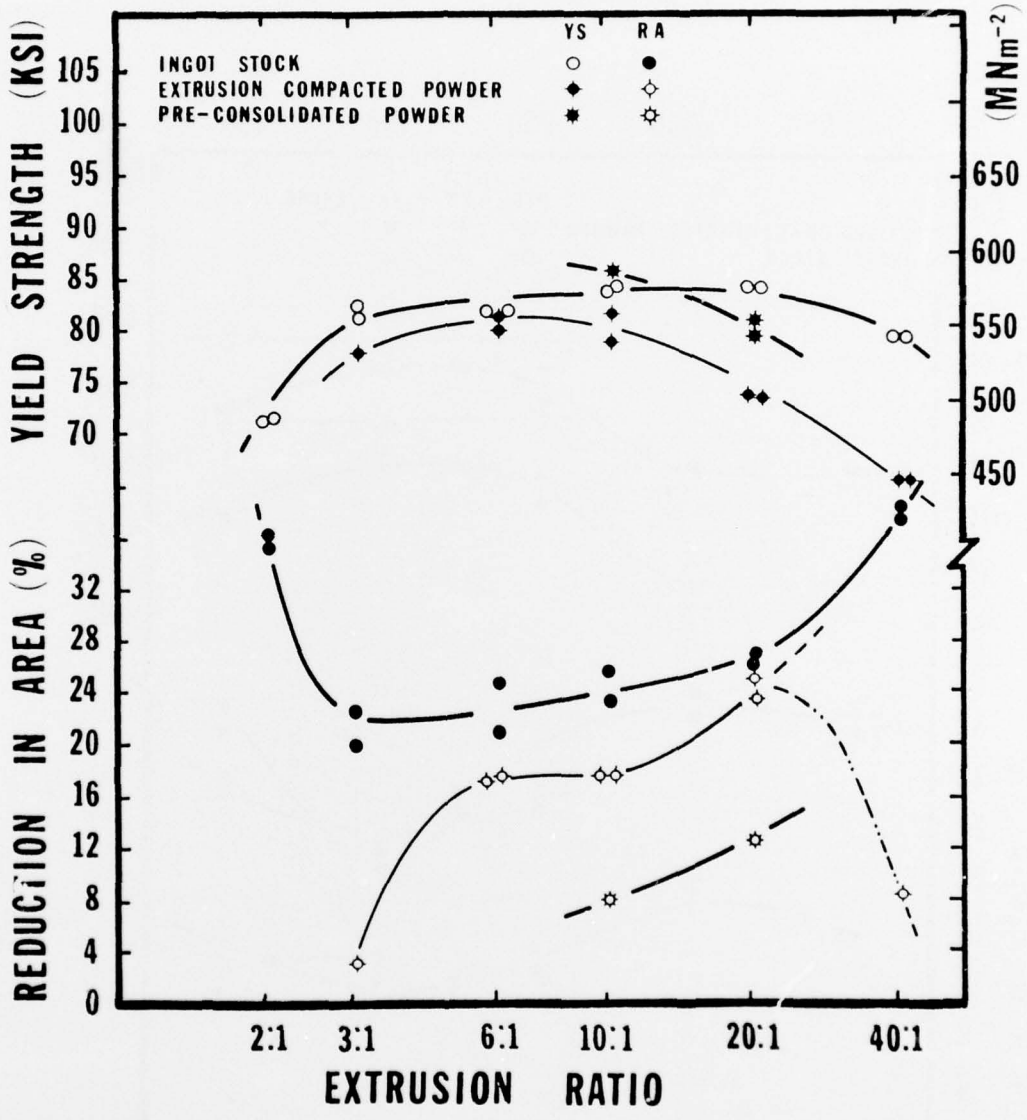

Figure 14a. Variation of yield strength and reduction in area in both ingot and powder product with extrusion ratio.

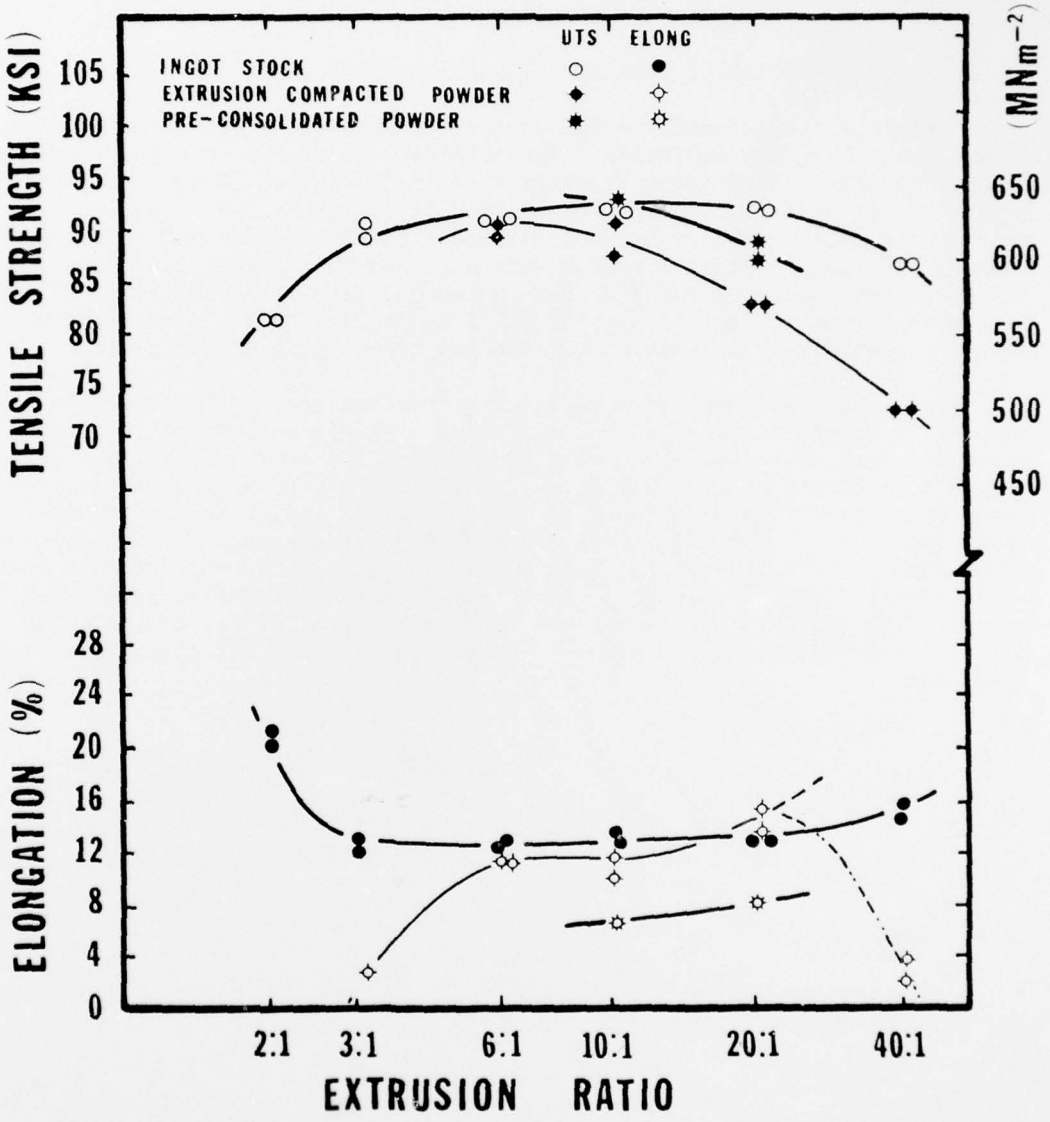

Figure 14b. Variation of ultimate tensile strength and elongation in both ingot and powder product with extrusion ratio.

APPENDIX

DESIGN OF CANS AND EVACUATION PROCEDURE

Containers to encapsulate the powder for subsequent extrusion were fabricated from tubing and bar stock. The outside wall of the containers were produced from commercially pure aluminum tube of 3.0 inches (0.076m) outside diameter. Nose and tail caps 1.0 inches (0.025m) thick by 3.0 inches (0.076m) diameter and a filling-evacuation tube of annealed 6061 aluminum 8 inches (0.2m) long by 1/2 inch (0.013m) diameter were utilized to complete the container with all components welded together. The containers were leak-checked by filling them with air at 160 psi (1.1×10^6 Nm^{-2}) and holding them under water while rotating them slowly to examine the adhered or escaping air bubbles.

After the containers were verified to be leak tight, powder was poured into them in an argon-filled glove box, yielding a poured density of approximately 60 percent. Each container was closed by covering the entry tube with a section of vacuum hose clamped shut. Subsequently, the container was evacuated at room temperature to 10^{-4}mm of mercury ($\simeq 1 \times 10^{-2}$ Nm^{-2}) and, with the vacuum pump operating continuously, the container was placed in a small furnace which was preheated to 700°F (644°K) and held for two hours. This procedure was intended to drive-off absorbed volatile contaminants. The container of powder was then removed from the furnace and allowed to cool to room temperature. It was sealed-off by crimp welding of the evacuation tube while the evacuation system was still operating.

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